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AUTHORITY

USNWC ltr, 30 Aug 1974

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IDEP FORM 12-11-62 IDEP Q4238 **SPECIFICATION** 1 of 1

AD846573

D0722

1. COMPONENT/PART NAME PER GENERIC CODE	2. PROGRAM OR WEAPON SYSTEM	3. DATE OF:	DAY	MO	YR
Propulsion Parts & Materials, Solid Fuel Engines, Propellants,	Multiple	ISSUE	13	10	67
4. ORIGINATOR'S SPECIFICATION TITLE: Purchase Description - Ammonium Nitrate (Uncoated)	5. ORIGINATOR'S SPEC. NO. WS 7650	REVISION			
	6. SPECIFICATION IS: ☐ DRAFT ☐ PRELIMINARY ☒ FINAL				

7. THIS SPECIFICATION COMPLEMENTS REPORT NO:

8. TYPE OF SPECIFICATION

- ☒ (A) GENERAL PRODUCT REQUIREMENTS FOR A FAMILY OF PARTS - PROCUREMENT DOCUMENT
- ☐ (B) INDIVIDUAL DETAIL PARTS DOCUMENT; STDS BOOK PAGES - FOR PROCUREMENT
- ☐ (C) DETAIL INSPECTION, PROCESS CONTROL, AND/OR TEST PROCEDURES FOR SPECIFIC PARTS
- ☐ (D) PROCESS (PAINTING, WELDING, FINISHING, HEAT TREATING ETC.) APPLICABLE TO MANY PARTS
- ☐ (E) SPEC. FOR PERFORMANCE, RELIABILITY, AND/OR ENVIRONMENT FOR ASSEMBLIES, EQUIPMENTS, SUBSYSTEMS AND SYSTEMS
- ☐ (F) PERFORMANCE AND APPLICATION DATA FOR DESIGN ENG. USE ON PARTS - NOT FOR PROCUREMENT
- ☐ (G) OTHER (DETAIL IN 10.)

NOTE: WHEN MULTIPLE FUNCTIONS ARE COMBINED IN ONE DOCUMENT, CHECK ALL APPLICABLE SQUARES

9. APPLICABLE REFERENCED SPECIFICATIONS EXCEPT MIL-SPECS, STDS, OR OTHER UNIVERSALLY AVAILABLE DOCUMENTS.	ENCL	AVAIL ON REQ	NOT AVAIL.	PREVIOUSLY SUBMITTED AND FILED AS IDEP REPORT OR SPEC NUMBER
1964 Book of ASTM Std. Part 17			x	
49 CFR 71-78			x	

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13 SPEC. NO 565.70.00.00-X7-03

11. SIGNED	12. CONTRACTOR	SUBCONTRACTOR
R. S. Harper 1/16/68	NWC/CL	

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NOTICES PAGE

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Code Ident 30003 WS 7650

NAVAL AIR SYSTEMS COMMAND

DEPARTMENT OF THE NAVY

PURCHASE DESCRIPTION

AMMONIUM NITRATE (UNCOATED)

1. SCOPE.

1.1 Scope. This purchase description covers one grade of ammonium nitrate.

2. APPLICABLE DOCUMENTS.

2.1 The following document of the issue in effect on date of invitation for bids or request for proposal forms a part of this document to the extent specified herein.

STANDARDS

Military

MIL-STD-129 Marking for Shipment and Storage.

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

FSC 6810

2.2 Other publications. The following documents form a part of this document to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

American Society for Testing and Materials (ASTM)

1964 Book of ASTM Standards; Part 17	Test-Method ASTM D128-64, "Analysis of Lubricating Grease".

(ASTM Publications are published by the American Society for Testing and Materials, Philadelphia 3, Pennsylvania.)

Code of Federal Regulations

49 CFR 71-78	Interstate Commerce Commission Rules and Regulations for the Transportation of Explosive and Other Dangerous Articles

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington, D.C., 20360.)

3. REQUIREMENTS.

3.1 Preproduction sample. Unless otherwise specified (see 6.2), a preproduction sample shall meet all requirements of this document. The preproduction sample shall be prepared using the same methods and procedures proposed for production. Any production prior to acceptance of the preproduction sample shall be at the risk of the supplier.

3.2 Data. No data is required by this document or by referenced documents in section 2 unless specified in the contract or purchase order.

3.3 Compliance to documents. Ammonium nitrate shall conform to the requirements herein and to the applicable requirements of documents listed in section 2.

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3.4 Product characteristics and performance. When tested in accordance with 4.7 of this document, ammonium nitrate shall meet the following product characteristics and performance.

3.4.1 Chemical analysis. The chemical analysis of the material shall be specified in Table I.

Table I. Chemical Analysis

Constituents	Min	Max
Ammonium nitrate (NH_4NO_3), %	99.0	---
Moisture, %	---	0.50
Ash, %	---	0.10
Ether-soluble material, %	---	0.10
Water-insolubles, %	---	0.10
Soluble chlorides, as NH_4Cl, %	---	0.03
Acidity, as free HNO_3, %	---	0.02
Acidity, pH	5	7

3.5 Workmanship. The ammonium nitrate shall be uniform in quality, free from foreign materials, and shall be manufactured under conditions and procedures standard in the industry.

4. QUALITY ASSURANCE PROVISIONS.

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial laboratory acceptable to the Government. The Government reserves the right to perform any of the inspections set forth in this document where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.2 Lot. A lot shall consist of material produced at one plant with no change in formulation or process. If manufacture is by batch process, each batch shall constitute a lot. A batch shall be as defined in 6.3.

4.3 Acceptance sampling. The number of containers to be chosen at random for acceptance sampling shall be equal to the square root of the total number of containers in the lot. If the number thus obtained is not a whole number, the number of containers to be sampled shall be increased to the next higher whole number. In no case, however, shall the number of containers to be sampled be less than seven (unless there are less than seven containers in the lot, in which case, each container shall be sampled).

4.3.1 Primary sample. From each selected container, a sample shall be taken from three or more places throughout the container. The total weight of the samples taken from each container shall weigh at least 50 grams (gm). Each sample thus taken shall be mixed thoroughly, placed in a clean dry container, and labeled to identify the material name, original container designation, contract number, and lot number.

4.3.2 Composite sample. Each primary sample shall be subdivided to prepare a composite sample (not in excess of 350 gm). Primary material not used shall be returned to the primary sample container. After mixing the composite sample thoroughly, the composite sample shall be placed in a clean, dry container and sealed. The composite sample shall be identified with the material name, container designation, contract number, and lot number. All specified chemical tests shall be made on this composite sample representing the lot. Failure of the composite sample to pass all of the tests herein shall result in rejection of the lot represented.

4.4 Classification of tests. Inspection and testing of ammonium nitrate shall be classified as follows:

(a) Preproduction tests.

(b) Quality conformance tests.

4.5 Preproduction tests. Preproduction tests shall be conducted only on the preproduction sample and shall consist of all examinations and tests specified in 4.6.

4.6 Quality conformance tests. Quality conformance tests for acceptance of the ammonium nitrate shall consist of the following tests:

Constituents	Test
Ammonium nitrate (NH_4NO_3)	4.7.1
Moisture	4.7.2
Ash	4.7.3
Ether - soluble material	4.7.4
Water-insolubles	4.7.5
Soluble chlorides, as NH_4Cl	4.7.6
Acidity as free HNO_3	4.7.7
Acidity pH	4.7.8

4.7 Tests. The following procedures shall be used to determine that the requirements of this document have been met. Any proposed change in test procedures or equipment shall necessitate, before adoption, prior approval of the procuring activity. In case of dispute between the results from any proposed method or equipment and what is cited herein, the results using the methods and the equipment specified in this document shall prevail. Unless otherwise specified, all tests shall be run in duplicate. The average of the two results shall be taken as the test result.

4.7.1 Ammonium nitrate (NH_4NO_3). Add 25 milliliters (ml) of approximately 40 percent formaldehyde solution and a few drops of phenolphthalein indicator solution to 100 ml of freshly boiled distilled water in a 125-ml Erlenmeyer flask. Neutralize the solution with 0.15N sodium hydroxide (NaOH),

add a weighed portion of approximately 1 gm of sample (weighed to the nearest 0.01 gm), heat the mixture to 60 degrees centigrade (°C) (140 degrees Fahrenheit (°F)), then cool to room temperature. Titrate the solution with 0.15N NaOH to an end point which persists at least 30 seconds.

$$\text{Percent ammonium nitrate} = \frac{8.005(AB) - 1.49C}{W}$$

Where: A = Volume of sodium hydroxide used, ml.

B = Normality (N) of sodium hydroxide solution.

C = Ammonium chloride in sample (4.7.6), percent.

W = Weight of sample taken, gm.

4.7.1.1 Acceptance criteria. For the lot represented to pass the ammonium nitrate test, the value obtained for percent ammonium nitrate shall be not less than the value specified in 3.4.1.

4.7.2 Moisture determination.

4.7.2.1 Apparatus. The apparatus used for determination of moisture content of the sample shall be Aquameter, Model KF-2 or KF-3, Beckman Instruments, Inc., Fullerton, California, or approved equivalent. The Aquameter shall be prepared for operation as described in the technical manual furnished by the manufacturer (Beckman Instruments, Inc.). Use of an alternate equivalent item of equipment approved by the procuring activity will necessitate use of the specific technical manual prepared by the manufacturer.

4.7.2.2 Reagents.

(a) Karl Fischer reagent. Karl Fischer reagent must have a strength such that each milliliter of Karl Fischer reagent corresponds to 0.0014-0.0023 gm of water. Dilute 750 ml of commercially available stabilized Karl Fischer reagent (with water equivalent of 0.005-0.007 gm/ml) to 2000 ml with absolute

methanol (0.1 percent water, maximum). Mix well and allow to stand overnight before use. Determine the water equivalent (A) of this solution as follows:

1. Use sodium tartrate dihydrate ($Na_2C_4H_4O_6 \cdot 2H_2O$) as a primary standard (with a water content of 15.66 percent) for standardizing Karl Fischer reagent. If the water content value is in question, it may be determined by heating some of the salt at 150°C (302°F) for 3 hours. Should the value (as determined) differ from the theoretical value of 15.66, then the experimental value shall be used in the determination of water equivalent (A) of the Karl Fischer reagent; i.e., instead of the 15.66 in the formula below, the factor should be 10P where P is percentage moisture (as determined). Rapidly transfer 0.090-0.110 gm (weighed to the nearest 0.0001 gm) of reagent-grade $Na_2C_4H_4O_6 \cdot 2H_2O$ to the titration vessel.

2. Titrate to an end point in the same manner as with the sample. (See 4.7.2.3.)

3. Repeat the standardization procedure until three successive results agree within five parts per thousand.

4. If the indicated water equivalent (A) of the Karl Fischer reagent is less than 0.0014 gm of water per ml of Karl Fischer reagent, it may be due to the presence of too much water in the absolute methanol used. In this case, distill the methanol from metallic calcium or calcium hydride. Passing the methanol through a column of Molecular Sieves, Type 4A, may also reduce the water content of the methanol sufficiently. (Molecular Sieves are a product of the Linde Company, a division of Union Carbide Corporation, New York City, New York.)

$$\text{Water equivalent (A)} = \frac{(156.6)\ W}{V}$$

Where: A = Water equivalent of the Karl Fischer reagent, gm/ml.

W = Weight of $Na_2C_4H_4O_6 \cdot 2H_2O$ taken, gm.

V = Volume of Karl Fischer reagent used, ml.

(b) Water-in-methanol solution. The water-in-methanol solution should contain 0.0015-0.0020 gm of water per ml of solution. A good grade of commercial absolute methanol contains approxiamtely 0.0010 gm of water per ml of methanol. Water content can be adjusted by adding 1.0 gm of distilled water to 1000 ml of water-in-methanol solution to produce a change of 0.0010 gm per ml. Determine the relative strength of the water-in-methanol solution in terms of Karl Fischer reagent as follows:

1. Put about 50 ml of the anhydrous methanol used in 4.7.2.2 (a) into the titration beaker of the Aquameter. Add a slight excess of Karl Fischer reagent (4.7.2.2 (a)), then back titrate with water-in-methanol solution (4.7.2.2 (b)). Then run in an additional 5 to 8 ml of Karl Fischer reagent, read to the nearest 0.01 ml, and again back titrate with water-in-methanol solution (read to the nearest 0.01 ml). Repeat the addition and back titrating steps twice more to provide triplicate determinations of the equivalency ratio. Calculate the ratio (B) of the Karl Fischer reagent to that of the water-in-methanol solution. The range of the ratios calculated from the three titrations should not be greater than 0.04. If the range exceeds 0.04, continue making titrations until three ratios are obtained whose range does not exceed 0.04. Then determine the average ratio from all the ratios which have been obtained.

4.7.2.3 Procedure. Determine the moisture content of the sample by the Karl Fischer method using a direct-titration technique. Introduce to the titration beaker, through the opening in the diaphragm, approximately 10 gm of sample weighed to the nearest 0.001 gm. Close the opening, start the stirrer, and press the titration button to titrate with Karl Fischer reagent (4.7.2.2 (a)). When the indicator light glows, read the Karl Fischer buret to the nearest 0.01 ml. Where necessary, water methanol solution (4.7.2.2 (b)) may be used to back titrate.

$$\text{Percent moisture} = \frac{100A\ (V_{KF} - BV_{WM})}{W}$$

Where:
- A = Weight of water equivalent to 1.00 ml of Karl Fischer reagent, gm/ml.
- V_{KF} = Volume of Karl Fischer reagent titrant used, ml.
- V_{WM} = Volume of water-in-methanol solution titrant used, ml.
- B = Ratio of Karl Fischer reagent to that of water-in-methanol solution, ml/ml.
- W = Weight of sample taken, gm.

4.7.2.4 Acceptance criteria. For the lot represented to pass the moisture test, the value obtained for percent moisture shall be no greater than the value specified in 3.4.1.

4.7.3 Ash. Determine the amount of ash in accordance with the procedure given in Test Method D128-64, "Analysis of Lubricating Grease" (Part 17 of ASTM, page 54).

4.7.3.1 Acceptance criteria. For the lot represented to pass the ash test, the value obtained for percent ash shall be no greater than the value specified in 3.4.1.

4.7.4 Ether-soluble material. Dry a clean, 150-ml beaker in an ovem at 100±5°C (212±9°F) for approximately one hour, cool in a desiccator, and weigh

to the nearest 0.001 gm. Weigh to the nearest 0.001 gm, approximately 25 gm of the specimen and transfer to the thimble of a Soxhlet extractor containing anhydrous ether. Extract the specimen for approximately 2 hours. Save the extracted specimen for the water-insoluble determination (4.7.5). Evaporate the ether extract in the extraction flask to a volume of approximately 50 ml on a steam bath and transfer to the weighed beaker. Rinse out the extraction flask with several 5-ml portions of ether and add them to the main extract in the beaker. Cover the beaker with a ribbed watch glass and evaporate on a steam bath. After the solvent is removed, heat the beaker and residue in an oven at 100°C (212°F) for one hour, cool in a desiccator, and weigh to the nearest 0.001 gm.

$$\text{Percent ether-soluble material} = \frac{100A}{W}$$

Where: A = Weight of residue from sample, gm.

W = Weight of sample taken, gm.

4.7.4.1 Acceptance criteria. For the lot represented to pass the ether-soluble-material test, the value obtained for percent ether-soluble-material shall be no greater than the value specified in 3.4.1.

4.7.5 Water-insolubles. Using the ether-extracted specimen from 4.7.4, dissolve it in approximately 100 ml of distilled water. Weigh a dry, sintered-glass funnel to the nearest 0.001 gm, then filter the mixture carefully through the funnel in order that all undissolved residue may be collected. Dry the funnel and residue for one hour in an oven at 105 ± 5°C (221 ± 9°F), allow to cool to room temperature in a desiccator, then reweigh to the nearest 0.001 gm.

$$\text{Percent water-insolubles} = \frac{(A-B)\ 100}{W}$$

Where: A = Weight of funnel plus residue, gm.

B = Weight of funnel empty, gm.

W = Weight of sample taken in 4.8.4, gm.

4.7.5.1 Acceptance criteria. For the lot represented to pass the water-insolubles test, the value obtained for percent water insolubles shall be no greater than the value specified in 3.4.1.

4.7.6 Soluble chlorides, as NH_4Cl. Place approximately 50 ml of chloride-free distilled water in a porcelain evaporating dish. Add 5 gm of sample (weighed to the nearest 0.0001 gm). Acidify the solution with approximately 2 ml of 3N nitric acid. Titrate the solution with an excess of 0.1N silver nitrate. Add a few drops of ferric alum indicator solution and, while swirling, titrate the excess silver nitrate with 0.1N ammonium thiocyanate. The end point is taken as the first permanent pink or red color observed.

$$\text{Percent ammonium chloride} = \frac{(AB-CD)\ 5.35}{W}$$

Where: A = Volume of silver nitrate used, ml.

B = Normality of silver nitrate solution.

C = Volume of ammonium thiocyanate solution used, ml.

D = Normality of ammonium thiocyanate solution.

W = Weight of sample taken, gm.

4.7.6.1 Acceptance criteria. For the lot represented to pass the soluble-chloride test, the value obtained for percent NH_4Cl shall be no greater than the value specified in 3.4.1.

4.7.7 Acidity, as free HNO_3. Dissolve approximately 100 gm of sample (weighed to the nearest 0.01 gm) in 400 ml of distilled water. Filter, add methyl red indicator, and titrate with 0.1N sodium hydroxide (NaOH). Calculate the acidity as follows:

$$\text{Acidity, as percent nitric acid} = \frac{6.3 \times N \times V}{W}$$

Where: N = Normality of NaOH solution.
V = Volume of NaOH solution used, ml.
W = Weight of sample taken, gm.

4.7.7.1 Acceptance criteria. For the lot represented to pass the acidity test, the value obtained for percent free HNO_3 shall be no greater than the value specified in 3.4.1.

4.7.8 Acidity, pH. The acidity, pH, specified in 3.4.1 shall be determined in accordance with 4.7.8.2.

4.7.8.1 Apparatus. Electrometric pH apparatus, or equal.

4.7.8.2 Procedure. Dissolve approximately one gm of sample (weighed to the nearest 0.01 gm) in 100 ml of freshly boiled distilled water contained in a 200-ml beaker. Determine the pH of the solution electrometrically using a pH meter.

4.7.8.3 Acceptance criteria. For the lot represented to pass the acidity (pH) test, the value obtained shall be within the range shown in 3.4.1.

4.8 Packing and marking. Determine that all packing and marking conforms to section 5 of this document.

5. PREPARATION FOR DELIVERY.

5.1 Preservation and packaging. Not applicable (unless specified in the contract or purchase order).

5.2 Packing.

5.2.1 Level A. Not applicable.

5.2.2 Level B. Not applicable.

5.2.3 Level C. The material shall be packed as directed in the contract to afford protection against damage during direct shipment from the supply source to the first receiving activity for immediate use. Containers shall comply with common carrier regulations applicable to the mode of transportation to be used. (See 6.2.)

5.3 Marking.

5.3.1 Special markings. The shipping containers shall be marked in accordance with the Code of Federal Regulations 49 CFR 71-78.

5.3.2 Normal markings. In addition to the markings required by contract or purchase order, unit packages and shipping containers shall be marked in accordance with the requirements of MIL-STD-129.

6. NOTES.

6.1 Intended use. The ammonium nitrate described in this document is intended for use as an ingredient in ammonium-nitrate-based solid propellants.

6.2 Ordering data. Procurement documents should specify the following:

(a) Title, number and date of this document.

(b) Whether a preproduction sample is required (see 3.1).

(c) Type and size of shipping container (see 5.2.3).

6.3 Definition.

6.3.1 Batch. A batch is defined as that quantity of material which has been subjected to one or more chemical or physical processes (or combinations thereof) intended to produce a desired product having substantially uniform characteristics. The final step in the processing must have treated the entire contents of the batch at one time.

6.4 Safety and health warning. When the use of any chemical is specified herein, suitable safety and health precautions should be observed.

6.5 Acceptable product. An acceptable product under this document is ammonium nitrate, Product Specification N-9 manufactured by Spencer Chemical Company, Kansas City, Missouri.

Custodian:
NASC 52021E

Preparing Activity:
NWC/China Lake, California